(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 456 044 A2**

(12) **EUROPEAN PATENT APPLICATION**

(21) Application number: **91106709.8**

(51) Int. Cl.5: **D04H 1/42**

(22) Date of filing: **25.04.91**

(30) Priority: **04.05.90 US 518904**
13.12.90 US 626817

(43) Date of publication of application:
13.11.91 Bulletin 91/46

(84) Designated Contracting States:
AT BE CH DE DK ES FR GB GR IT LI LU NL SE

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(54) **Three dimensional polymer webs and methods of making same.**

(57) Three-dimensional nonwoven webs comprise at least one layer consisting of drawn and unoriented thermoplatic fibers formed from a blend of polypropylene and polybutylene, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene. The blend can be a blend of a homopolymer of polypropylene and a homopolymer of polybutylene: a homopolymer of polypropylene and a copolymer of polybutlene: a copolymer of polypropylene and a homopolymer of polybutylene: and a copolymer of polypropylene and a copolymer of polybutylene. In addition, ternary blends comprising polypropylene, polybutylene and a terpolymer (propylene, ethylene, and 1-butene) are disclosed. The resulting nonwoven webs have high strength, toughness, and tear resistance.

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This invention relates generally to three-dimensional nonwoven webs and methods of making same.

Nonwoven webs formed of thermoplastic fibers are well known in the art and have found uses in a variety of applications. In one application, such nonwoven webs are formed by melt-blowing and are used as disposable industrial wipers. Such industrial wipers find application in numerous manufacture and maintenance facilities where personnel find it necessary to wipe up oil, grease, and water from a variety of surfaces. One such wiper made of melt-blown polypropylene fibers is manufactured and sold by Kimberly-Clark Corporation, the assignee of the present invention under the trademark Kimtex®.

Melt-blown nonwoven wipers of polypropylene thermoplastics fibers have advantage over cloth wipers in being cost effectively disposable with similar wiping characteristics as compared to cloth. Particularly, industrial wipers must be able to quickly pick up spilled liquids, both oil based and water based, and leave a clean, streak free surface. In addition, the wipers must have sufficient capacity to hold such liquids within the wiper structure until it is desired to remove the liquid by pressure such as by wringing.

Nonwoven melt-blown industrial wipers formed from polypropylene in the past have performed adequately in terms of their wiping characteristics, particularly with respect to oil and, when treated with a sufactant, with respect to water. Moreover, nonwoven industrial wipers made of polypropylene fibers have exhibited resistance to most commercially available solvents.

A superior three dimensional nonwoven melt-blown wiper, however, could be achieved by increasing strength, increasing toughness, and increasing tear resistance. A three dimensional nonwoven web is described in U.S. Patent No. 4,741,941, to Englebert, which is incorporated herein by reference. However, the web described in Englebert does not teach the increased strength which characterizes a superior three dimensional nonwoven melt-blown wiper.

It is therefore an object of the present invention to provide a three dimensional nonwoven web consisting of thermoplastic fibers, which web will display improved strength, toughness, and tear resistance over three dimensional melt-blown webs formed of polypropylene and polyethylene fibers.

It is likewise an object of the present invention to provide an industrial wiper comprising a three dimensional nonwoven melt-blown web consisting of thermoplastic fibers which will provide improved strength, toughness, and tear resistance over three dimensional melt-blown wipers formed of polypropylene and polyethylene fibers.

It is also an object of the present invention to provide a laminate material comprising at least one three dimensional layer consisting of thermoplastic fibers which will provide improved strength, toughness, and tear resistance over laminate materials which contain at least one three dimensional layer formed of polypropylene and polyethylene fibers.

It is a further object of the present invention to provide an industrial wiper formed from a laminate material comprising at least one three dimensional layer consisting of thermoplastic fibers which will provide improved strength, toughness, and tear resistance over industrial wipers formed from laminate materials which contain at least one three dimensional layer formed of polypropylene and polyethylene fibers.

The foregoing objectives are obtained by nonwoven webs consisting of drawn and unoriented thermoplastic fibers formed from blends of polypropylene and polybutylene including blends of homopolymers of polypropylene and homopolymers of polybutylene; from blends of homopolymers of polypropylene and copolymers of polybutylene; from blends of copolymers of polypropylene and homopolymers of polybutylene; and from blends of copolymers of polypropylene and copolymers of polybutylene.

In addition, the foregoing objects can be obtained by nonwoven webs consisting of drawn and unoriented thermoplastic fibers formed from a ternary blend comprising polypropylene, polybutylene and a terpolymer comprising propylene, ethylene, and 1-butene.

The invention therefore provides, according to a first preferred aspect, a material comprising at least one layer consisting of drawn and preferably unoriented thermoplastic fibers formed from a blend comprising polypropylene and polybutylene, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene. This material is preferably a three dimensional nonwoven web according to independent claim 1, a three dimensional nonwoven web according to independent claim 2 or 4 or a laminate material according to independent claim 3. The invention also provides a method for forming such material comprising the steps of:

a. mixing polypropylene and polybutylene to form a blend, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene;
b. heating the blend to form a melt;
c. extruding the melt through a die to form thermoplastic fibers;
d. initially drawing the fibers to a ratio of greater than 14 to 1 and
e. depositing the fibers onto a forming surface to form a first layer.

This method is preferably used in a method for forming a layer of three dimensional nonwoven web

according to independent claim 8 and a method for forming a laminate material according to independent claim 9. Further advantageous features and details of the material and the method are evident from the dependent claims, the description, examples and drawings.

The invention also provides according to a second preferred aspect, a material comprising at least one layer consisting of drawn thermoplastic fibers formed from a ternary blend comprising polypropylene, polybutylene and a terpolymer, wherein the ternary blend by weight is from 10% to 30% polybutylene. This material is preferably a three dimensional nonwoven web according to independent claim 16, a three dimensional nonwoven wiper according to independent claim 17 or 19, or a laminate material according to independent claim 18. The invention also provides a method for providing such a material comprising the steps of:

a. mixing polypropylene, polybutylene and a terpolymer to form a ternary blend, wherein the ternary blend by weight is from 10% to 30% polybutylene
b. heating the ternary blend to form a melt
c. extruding the melt through a die to form thermoplastic fibers
d. initially drawing the fibers to a ratio of greater than 14 to 1 and
e. depositing the fibers onto a forming surface to form a first layer.

This method is preferably used in a method for forming a layer of three dimensional nonwoven web according to independent claim 23 and a method for forming a three dimensional nonwoven wiper according to claim 24 or 25.

Further advantageous features and details of the material and the method are evident from the dependent claims, the description, examples and drawings.

In connection with the present invention, "drawn thermoplastic fibers" refers to fibers that are drawn to a ratio of greater than 14 to 1 (generally greater than 50 to 1) in the forming process. Also in connection with the present invention, "unoriented thermoplastic fibers" refers to fibers which solidify in a relaxed condition (not under tension). Such drawn and unoriented fibers are characteristically formed by melt-blowing techniques as well as other fiber forming techniques such as melt-spraying.

More particularly, the foregoing objectives are realized by a 3-dimensional nonwoven melt-blown material especially a web consisting of drawn and unoriented thermoplastic fibers formed by melt-blowing a blend comprising polypropylene and polybutylene. Particularly, the three dimensional blend of polypropylene and polybutylene is from 90-70% by weight of polypropylene and from 10-30% by weight of polybutylene. More particularly, in the case of a nonwoven wiper, the three dimensional blend is preferred to be from 85% to 75% by weight of polypropylene and from 15% to 25% by weight of polybutylene.

In connection with the present invention, the prior art European Patent Application No. 89303407.4 * of Don & Low Ltd. has disclosed the usefulness of blending polypropylene and polybutylene to form fibers, tapes, and films where the thermoplastic fibers, tapes, and films are drawn to a ratio of at least 8:1 to produce an oriented molecular structure. Particularly, the Don & Low reference discloses thermoplastic fibers, tapes, and films that have improved strength as measured by tenacity which is the maximum stress they can resist without rupture. Such thermoplastic fibers, tapes, and films result from blending up to 10% by weight of polybutylene with polypropylene with from 2% to 4% by weight of polybutylene being preferred. The Don & Low reference, however, teaches that "it has been found that if the polybutylene proportion is increased beyond ten percent there is little if any increase in strength [tenacity]". Also the Don & Low reference does not teach improved elongation. As will be demonstrated hereinbelow the nonwoven melt-blown and melt-spray webs of the present invention result from a blend of polypropylene and polybutylene with the polybutylene in excess of 10%. Moreover, the thermoplastic fibers of the present invention are drawn to a ratio of substantially greater than 14 to 1 and are not oriented as a result of the melt-blowing or melt-spraying process.

Fig. 1 is a schematic diagram showing showing machinery for producing a three dimensional nonwoven melt-blown web in accordance with the present invention.

While the invention will be described in connection with a preferred embodiment and procedure, it will be understood that we do not intend to limit the invention to that embodiment or procedure. On the contrary, we intend to cover all alternatives, modifications, and equivalents as may be included within the spirit and scope of the invention as defined by the appended claims.

Turning to Fig. 1 there is shown a web forming machine 10 for forming a melt-blown web 12 made up of a number of layers or melt-blown fibers 50. The machine 10 includes eight identical extruders 14A-H with corresponding hoppers 16A-H for receiving thermoplastic resin pellets. The extruders 14A-H include internal screw conveyors which are driven by motors 15A-H. The extruders 14A-H are heated along their lengths to

* publication no. 337 662

the melting temperature of the thermoplastic resin pellets to form a melt. The screw conveyors driven by motors 15A-H force the thermoplastic material through the extruders into attached delivery pipes 20A-H which are connected to die heads 22A-H, each having a die width 25.

Die head 22A, for example, comprises a die tip which has a die opening or orifice (not shown). The die tip may be recessed, flush, or stick out. Hot fluid, usually air, is supplied to the die tip via pipes 32 and 34 (Fig. 1) which terminate in channels adjacent to the orifice of the die tip.

As the thermoplastic polymer exists the die tip for each die head, the high pressure air attenuates and breaks up the polymer stream to form fibers at each die head. The fibers are then in accordance with the present invention quenched with a mist of water from spray nozzles 27A-H. The spray nozzles are located just below the orifice of the die tip and spray the fibers 50 with water at room temperature or slightly above at a flow rate of at least 0.02 liter/min/inch * width of die tip. Fiber quenching is conventional as demonstrated by United States Patent No. 3,959,421. If desired, a surfactant can be added to the fibers by adding the surfactant to the quenching water. It should also be noted that significantly improved toughness and tear resistance result only if the fibers are subjected to quenching.

Once the fibers have been quenched, they are deposited on a forming surface 38 to form the layered web. In the preferred embodiment of the present invention, the forming surface 38 is an open mesh wire surface into which the fibers are pulled in order to form the three dimensional layer. However, a number of alternative types of forming surfaces 38 may be utilized in order to produce the desired three dimensional layer. A variety of such forming surfaces are described in the Englebert patent and are well-known in the art.

A vacuum is drawn behind the forming surface 38 to draw the fibers onto the forming surface 38 during the process of melt-blowing. Separate vacuum chambers behind the forming surface 38 may be provided for each die head 22A-H. Once the fiber layers have been deposited on the forming surface 38 by the multiple die heads 22A-H, the web 12 is drawn from the forming surface 38 by withdrawal rolls 40 and 42. Embossing rolls 44 and 46 engage the web 12 after the withdrawal rolls to emboss the web with a pattern.

The foregoing description of the melt-blowing machine 10 is generally conventional and well known in the art as demonstrated by NRL Report 4364, "Manufacture of Super-Fine Organic Fibers:, by V.A. Wendt, E.L. Boon, and C.D. Fluharty; NRL Report 5265, "An Improved Device for the Formation of Super-Fine Thermoplastic Fibers", by K.D. Lawrence, R.T. Lukas, and J.A. Young; and United States Patent 3,849,241, issued November 19, 1974, to Buntin, et al. It also will be appreciated by one of ordinary skill in the art that a single head melt-blowing machine can be used instead of the multiple bank machine illustrated. It should also be appreciated that the web 12 may be comprised of a single layer, multiple layers which are all identical in composition, or multiple layers some of which layers are made in accordance with the present invention and some of which layers are conventional. Moreover, ordinary skill in the art will appreciate that fine adjustment of the equipment and process may be required to optimize performance and efficiency. Such fine adjustment can be accomplished by one of ordinary skill without undue experimentation.

In addition, the fibers and the resulting web can be formed by other fiber forming techniques including, for example, melt-spray techniques. Melt-spraying is a process for forming fibers from a fiber forming resin using compact spray head designs usually including one to four spray heads in a cluster. The equipment includes a die housing with a hydraulic chamber and a retractable piston assembly for releasing molten resin. As the molten resin is released, it is contacted and drawn by a primary air supply which completely surrounds the molten resin and contacts it at a predetermined angle. If additional drawing and attenuation of the newly formed fibers is desired, secondary fiberization air also may be utilized. The secondary fiberization air will most typically include at least two fluid streams which each impinge upon the resin/fibers at a second angle.

In more refined embodiments the fiberization air can also be angled such that it will spiral around the forming fibers. Additionally, the piston within the hydraulic chamber may be cycled on and off to interrupt the flow of the fiber forming resin thereby creating discrete pluralities of fibers.

Both melt-blowing and melt-spraying techniques produce fibers that are drawn and unoriented.

In accordance with the present invention, it has been found that an improved three dimensional nonwoven web of drawn and unoriented thermoplastic fibers can be formed by melt-blowing a blend of polypropylene and polybutylene to enhance certain properties in the melt-blown web 12, as compared with 100% three dimensional melt-blown polypropylene and polyethylene. Specifically, the melt-blown web 12 formed from melt-blowing a blend of polypropylene and polybutylene improves the strength, toughness (the elongation and absorbed energy), and tear resistance. Particularly, we have found that blends of from 90% to 70% polypropylene and from 10% to 30% polybutylene produce three dimensional nonwoven melt-blown

* 1 inch = 2.54 cm

webs with these improved characteristics. The advantages of the present invention can be realized by blending homopolymers of polypropylene and polybutylene; by blending copolymers of polypropylene and homopolymers or polybutylene; by blending homopolymers of polypropylene and copolymers of polybutylene; and by blending copolymers of polypropylene and copolymers of polybytylene. The copolymers of polypropylene may have ethylene in amounts ranging up to about 3% by weight. The copolymers of polybutylene may have ethylene in amounts ranging up to about 6% by weight.

In addition, the invention may be carried out by using ternary blends comprising polypropylene, polybutylene, and a terpolymer (propylene, ethylene, and 1-butene). The resulting three dimensional nonwoven webs have enhanced strength, toughness, and tear resistance.

Webs in accordance with the present invention can be formed at polymer throughputs of from 1 to 15 pounds * per inch of die head width per hour (pih). Once the melt-blown web has been formed, the material may be bonded or unbonded. The bond pattern may be a line pattern, a weave pattern, or a point pattern, but the point pattern, with bonding occurring within certain discrete areas on the material, is preferred. The bonding may be accomplished by ultrasonic heating, by use of an adhesive, or by embossing with heated rolls. In connection with the present invention, most webs described in the following examples were heat embossed with a weave pattern, such as that shown in United States Patent Des. 264,512, or with a dot pattern. The resulting bonded webs had a bonded area of about 18% of the total area when embossed with the weave pattern and of 5% - 18% when embossed with the dot pattern.

In carrying out the present invention, the blends and ternary blends were formed by simply mixing the thermoplastic pellets in the proper weight proportions before being added to the hoppers 16A-H. The terpolymers were polymerized in the desired proportions during manufacture.

Three dimensional nonwoven webs formed from blends, ternary blends, terpolymers, and terpolymer blends utilizing the present invention were made and tested in accordance with the following examples which illustrate the invention.

Example 1

A melt-blowing machine similar to that shown in Figure 1 was used to form a control web, Sample 59, from 100% polypropylene and four additional webs, Samples 61, 62, 64, and 66, from blends of 90% polypropylene and 10% polybutylene, 85% polypropylene and 15% polybutylene, 80% polypropylene and 20% polybutylene, and 70% polypropylene and 30% polybutylene, respectively. The polypropylene used for the control web, Sample 59, was a homopolymer with a melt flow of 800g/10min (ASTM D1288, Condition L, (230° C, 2160g weight)) and a narrow molecular weight distribution. Such a polypropylene homopolymer is manufactured by Himont U.S.A., Inc of Wilmington, Delaware and designated Valtec HH442H. The polypropylene used for the inventive melt-webs, Samples 61, 62, 64 and 66, was also Valtec HH442H. The polybutylene used for Samples 61, 62, 64, and 66 was Duraflex DP-8910, which is an ethylene copolymer (6% ethylene) and is manufactured by Shell Chemical Company of Houston, Texas. The melt-blowing machine was set up in accordance with the following process conditions for Samples 59, 61, 62, 64, and 66:

Machine Configuration:

A. recess die tip
B. single bank
C. water quench with surfactant

1 lb. = .454 kg
1 in. = 2.54 cm

Set Points:

		Sample 59	Samples 61- 66
Extruder Barrel Pressure	psi*	500	500
Primary Air Pressure	psi	2.4	2.2
Primary Air Temperature	°F	583	565
Forming Distance	in	6.5	6.0

1 psi = *0.069 bar

Measured Variables:

Die Tip Pressure	psi	21	15
Die Tip (Melt) Temperature	°F	497	489

In order to provide an accurate comparison between conventional Sample 59 and inventive Samples 61, 62, 64, and 66, the actual data for the conventional web, Sample 59, and the inventive webs, Samples 61, 62, 64, and 66, has been normalized to a basis weight of 1 gsm. Both the actual data and the normalized data which show the characteristics of Samples 59, 61, 62, 64, and 66 are set out in Table 1 below.

TABLE 1 -- COMPARISON WITH 100% POLYPROPYLENE

POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS IN THREE DIMENSIONAL MELTBLOWN

Sample Number	Composition	Basis Weight gsm	GRAB TENSILE Load MD g	Load CD g	Energy MD kg-mm	Energy CD kg-mm	Elong MD mm	Elong CD mm	TRAP TEAR Load MD g	TRAP TEAR Load CD g	NORMALIZED GRAB TENSILE Load MD g/gsm	NORMALIZED GRAB TENSILE Load CD g/gsm	NORMALIZED TRAP TEAR Load MD g/gsm	NORMALIZED TRAP TEAR Load CD g/gsm
59	100% PP	42	1344	1453	26	46	30	50	245	227	32	35	6	5
61	90:10 PP:PB	25	1544	1444	73	62	75	72	454	368	63	59	18	15
62	85:15 PP:PB	41	1839	1562	87	70	76	73	558	327	45	38	14	8
64	80:20 PP:PB	25	1880	1775	53	60	46	55	545	381	76	72	22	15
66	70:30 PP:PB	26	2393	1525	52	41	30	42	849	420	91	58	32	16

In connection with the characteristics reported in Table 1, the basis weight was measured in accordance with Federal Test Method 191A-5 and expressed in grams per square meter (gsm). The bulk was measured in accordance with the Ames Method and expressed in millimeters (mm). The grab tensile strength was measured in accordance with method 5100, Federal Test Method 191A and expressed in grams (g), kilogram-millimeters (kg*mm), and millimeters (mm). The trapezoid tear was measured in accordance with Method 5135, Federal Test Method 191 and expressed in grams (g).

In order to measure strength and toughness, the various samples were subjected to tensile testing in the machine direction and the cross-machine direction. Toughness is determined by the amount of energy that the material will absorb prior to failure. The peak energy is the amount of energy the material will absorb until the peak load is achieved. The fail energy is the amount of total energy the material will absorb until it finally fails by separating. Particularly as the stress to which the web is subjected is increased, the web begins stretching or elongating. At some point, the web reaches a peak loading at which failure begins and additional stressing does not increase the loading on the web. As the material begins failing, further elongation results under decreasing loading. The amount of peak energy the material experiences is the integral of the load v. elongation curve for the web from 0 load to the peak of the load. The amount of failed energy is the integral of the load v. elongation curve for the web from the initial 0 load until rupture occurs and the load again returns to 0. Both peak energy and fail energy give a good indication of the toughness of a web. Peak strength is determined by measuring the maximum load achieved before the web begins to fail.

Consequently, it can be seen from Table 1 that Samples 61 - 66, made in accordance with the present invention, all demonstrate a substantial increase in the peak load and energy over those of the prior art 100% three dimensional polypropylene melt-blown web of Sample 59. Particularly, Samples 61- 66 have an increase in peak load in the cross-machine direction of between 15% and 78% over the control Sample 59. Additionally, Samples 61 - 66 have an increase in peak energy in the machine direction of between 100% and 215% over the control Sample 59. Accordingly, the inventive webs, Samples 61 - 66, all demonstrate the increased strength and toughness of the three dimensional nonwoven webs prepared in accordance with the present invention.

While toughness measures a web's resistance to initial tearing, trapezoid tear measures the web's resistance to the propagation of a tear after an initial tear. As can be seen again from Table 1, Samples 61 - 66 show between a 85% and 247% increase in their tear resistance over that of the control Sample 59.

Example 2

Inventive webs, Samples 61 - 66, were next compared to a control web, Sample 2, formed from 100% polyethylene. The polyethylene used for the control web, Sample 2, was a copolymer (with 1-octene) with a melt index of 135 (ASTM D1288, Condition E, (190° C, 2160g weight)) and a narrow molecular weight distribution. Such a polyethylene homopolymer is manufactured by Dow Chemical, Inc. of Wilmington, Delaware and designated ASPUN® 6814A. The melt-blowing machine was set up in accordance with the following process conditions for Sample 2:

Machine Configuration:

A. recess die tip
B. single bank
C. water quench with surfactant

Set Points:		
Extruder Barrel Pressure	psi	504
Primary Air Pressure	psi	4.2
Primary Air Temperature	°F	507
Forming Distance	in	7.5
Measured Variables:		
Die Tip Pressure	psi	139
Die Tip (Melt) Temperature	°F	507

As with Example 1, the actual data for the conventional web, Sample 2, and the inventive webs, Samples 61, 62, 64, and 66, has been normalized to a basis weight of 1 gsm. Both the actual data and the normalized data which show the characteristics of Samples 2, 61, 62, 64, and 66 are set out in Table 2 below.

TABLE 2 -- COMPARISON WITH 100% POLYETHYLENE

POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS IN THREE DIMENSIONAL MELTBLOWN

Sample Number	Composition	Basis Weight gsm	GRAB TENSILE Load MD g	GRAB TENSILE Load CD g	GRAB TENSILE Energy MD kg-mm	GRAB TENSILE Energy CD kg-mm	GRAB TENSILE Elong MD mm	GRAB TENSILE Elong CD mm	TRAP TEAR Load MD g	TRAP TEAR Load CD g	NORMALIZED GRAB TENSILE Load MD g/gsm	NORMALIZED GRAB TENSILE Load CD g/gsm	NORMALIZED TRAP TEAR Load MD g/gsm	NORMALIZED TRAP TEAR Load CD g/gsm
2	PE	40	985	908	44	55	67	97			25	23		
61	90:10 PP:PB	25	1544	1444	73	62	75	72	454	368	63	59	18	15
62	85:15 PP:PB	41	1839	1562	87	70	76	73	558	327	45	38	14	8
64	80:20 PP:PB	25	1880	1775	53	60	46	55	545	381	76	72	22	15
66	70:30 PP:PB	26	2393	1525	52	41	30	42	849	420	91	58	32	16

It can be seen from Table 2 that Samples 61 - 66, made in accordance with the present invention, all demonstrate a substantial increase in the peak load and energy over those of the prior art 100% three dimensional polyethylene melt-blown web of Sample 2. Particularly, Samples 61 - 66 have an increase in peak load in the machine direction of between 57% and 143% over the control Sample 2. Additionally, Samples 61 - 66 have an increase in peak energy in the machine direction of between 18% and 98% over

the control Sample 2. Accordingly, the inventive webs, Samples 61 - 66, all demonstrate the increased strength and toughness of the three dimensional nonwoven webs prepared in accordance with the present invention.

Example 3

In order to determine the effectiveness of the ternary blends of the present invention, Samples 4, 6, 7 and 8 were prepared in accordance with the present invention for comparison with control Samples 2 and 59. Samples 4 and 6 were formed from a blend of 50% polypropylene (homopolymer; Himont HH442H) and 50% terpolymer (formed from three monomers, namely propylene, ethylene, and 1-butene), which was an experimental polymer manufactured by Himont U.S.A., Inc. of Wilmington, Delaware, identified by the designation 9582-35-1, having a melt flow rate of 45 g/10 min. Samples 7 and 8 are examples of a ternary blend of polymers in accordance with the present invention. Particularly, Samples 7 and 8 were formed from a blend of 45% polypropylene (homopolymer; Himont HH442H), 45% terpolymer (same terpolymer as present in Samples 4 and 6), and 10% polybutylene (Shell DP8910).

Samples 4, 6, 7, and 8 were formed by melt-blowing in accordance with the following parameters:

Machine Configuration:

A. recess die tip
B. single bank
C. water quench with surfactant

Set Points:		Samples 4/6	Samples 7/8
Primary Air Pressure	psi	3	2.5
Primary Air Temperature	°F	623	635
Forming Distance	in	7	7
Measured Variables:			
Die Tip Pressure	psi	125	128
Die Tip (Melt) Temperature	°F	579	557

As with the previous examples, the actual data for the inventive webs, Samples 4, 6, 7, and 8, has been normalized to a basis weight of 1 gsm. Both the actual data and the normalized data which show the characteristics of Samples 2, 4, 6, 7, 8, and 59 are set out in Table 3 below.

TABLE 3 -- COMPARISON WITH TRIBLEND PP:PB:TERPOLYMER POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS IN THREE DIMENSIONAL MELTBLOWN

Sample Number	Composition	Basis Weight gsm	GRAB TENSILE Load MD g	Load CD g	Energy MD kg-mm	Energy CD kg-mm	Elong MD mm	Elong CD mm	NORMALIZED GRAB TENSILE Load MD g/gsm	Load CD g/gsm
7 & 8	45:45:10 PP:TERP:PB	41	1871	1738	85	98	63	85	46	42
4 & 6	50:50 PP:TERP	41	2395	1841	53	67	28	51	59	45
59	100% PP	42	1344	1453	26	46	30	50	32	35
2	100% PE	40	985	908	44	55	67	97	25	23

It can be seen from Table 3 that although Samples 4 and 6, made in accordance with the present invention, demonstrate a substantial increase in the peak load and energy over those of the prior art three dimensional melt-blown webs of Samples 2 and 59, the polypropylene/terpolymer blend has a decreased elongation, which is an important characteristic of toughness. To the contrary, Samples 7 and 8 demonstrate increased strength, toughness, and elongation. Particularly, Samples 7 and 8 have an increase in peak load in the machine direction of between 39% and 90% over the control Samples 2 and 59. Additionally, Samples 7 and 8 have an increase in peak energy in the machine direction of between 93% and 227% over the control Samples 2 and 59, an increase in elongation of between 70% and 100%

compared with control Sample 59, and a comparable elongation with that of control Sample 2. Accordingly, the inventive ternary blend webs. Samples 7 and 8, demonstrate the increased strength and toughness of the three dimensional nonwoven webs prepared in accordance with the present invention.

Example 4

In order to test the effectiveness of the three dimensional web of the present invention as a laminate material, Samples 53, 56, 213, C. D were prepared in accordance with the present invention. Each of these samples was bonded to an intermediate layer of material which was not the three dimensional web of the present invention.

Samples 55 and 207 were also prepared to show the effectiveness of the three dimensional web of the present invention as a laminate material in situations where the three dimensional web will be directly bonded to another three dimensional web, without any intermediate layer.

Sample 53 was formed from a blend of 60% polyethylene (Dow 6814) and 40% polypropylene. The polypropylene used for Sample 53 was a homopolymer with a melt flow of 400g/10min (ASTM D1288, Condition L, (230°C, 2160g weight)) and a narrow molecular weight distribution. Such a polypropylene homopolymer is manufactured by Himont U.S.A., Inc. of Wilmington, Delaware and designated HH441. Sample 56 was formed from a blend of 75% polyethylene (Dow 6814) and 25% polypropylene (Himont HH441). As an intermediate material to which the laminate materials of Samples 53 and 56 were bonded, Intermediate Layer X was formed from a blend of 60% pulp (IPSS) and 40% ethylene vinyl acetate copolymer (Exxon "Escorene").

Sample 213 was formed from a blend of 25% polybutylene (Shell DP8910) and 75% polypropylene (Himont HH442H) and was bonded to an intermediate material "X", formed from a blend of 70% pulp (IPSS) and 30% ethylene vinyl acetate copolymer (Exxon "Escorene").

Sample C was formed from a blend of 80% polypropylene (Himont HH442H) and 20% polybutylene (Shell DP8910) which was bonded to an intermediate material "X", formed from a blend of 30% pulp (Weyerhauser NF105) and 70% polymer blend (comprised of 85% polypropylene (Himont HH442H) and 15% polybutylene (Shell DP8910)).

Sample D was formed from a blend of 80% polypropylene (Himont HH442H) and 20% polybutylene (Shell DP8910) which was bonded to an intermediate material "X", formed from a blend of 50% pulp (Weyerhauser NF105) and 50% polymer blend (comprised of 85% polypropylene (Himont HH442H) and 15% polybutylene (Shell DP8910)).

Sample 55 was formed from a blend of 75% polyethylene (Dow 6814) and 25% polypropylene (Himont HH442H). Sample 207 was formed from a blend of 80% polypropylene (Himont HH442H) and 20% polybutylene (Shell DP8910).

All the webs were heat embossed with a conventional bonding pattern.

Samples 53, 56, 213, C.D. 55, and 207 were formed by melt-blowing in accordance with the following parameters:

Machine Configuration:

A. recess die tip
B. single bank
C. water quench with surfactant

		3D Layer Sample 53	Intermediate Layer X
Primary Air Pressure	psi *	1.5	1.6
Primary Air Temperature	°F	512	404
Forming Distance	in	6.5	9
Primary Air Flow	scfm	168	
Measured Variables:			
Die Tip Pressure	psi	132	
Die Tip (Melt) Temperature	°F	589	381
Bonding:			
Pattern Roll Temperature			
Set Point	°F	180	
Actual Surface	°F	160	
Anvil Roll Temperature			
Set Point	°F	180	
Actual Surface	°F	170	
Pressure (Average)	psi	10	
Line Speed	fpm **	14	

		3D Layer Sample 56	Intermediate Layer X
Primary Air Pressure	psi		1.6
Primary Air Temperature	°F	545	404
Forming Distance	in		9
Primary Air Flow	scfm	400	

*1 psi = 0.069 bar
**1 fpm = 0.305 meters per minute

Measured Variables:			
Die Tip Pressure	psi	174	
Die Tip (Melt) Temperature	°F	538	381
Bonding:			
Pattern Roll Temperature			
Set Point	°F	180	
Actual Surface	°F	160	
Anvil Roll Temperature			
Set Point	°F	180	
Actual Surface	°F	170	
Pressure (Average)	psi	10	
Line Speed	fpm	14	

		3D Layer Sample 213	Intermediate Layer X
Primary Air Pressure	psi	2.3	5.0
Primary Air Temperature	°F	511	380
Forming Distance	in	7.5	
Primary Air Flow	scfm	66	
Measured Variables:			
Die Tip Pressure	psi		220
Die Tip (Melt) Temperature (and Triton X-102 surfactant quench)	°F	536	370
Bonding:			
Pattern Roll Temperature			
Set Point	°F	185	
Actual Surface	°F	160	
Anvil Roll Temperature			
Set Point	°F	190	
Actual Surface	°F	173	
Pressure (Average)	psi	12.5	
Line Speed	fpm	21	

		3D Layer Sample C	Intermediate Layer X
Primary Air Pressure	psi	1.6	6.3
Primary Air Temperature	°F	517	514
Forming Distance	in	7	18
Primary Air Flow	scfm		

Measured Variables:			
Die Tip Pressure	psi	79	117
Die Tip (Melt) Temperature (and Triton X-102 surfactant quench)	°F	495	514
Bonding:			
Pattern Roll Temperature			
Set Point	°F	220	
Actual Surface	°F		
Anvil Roll Temperature			
Set Point	°F	230	
Actual Surface	°F		
Pressure (Average)	psi	16	
Line Speed	fpm	22	

		3D Layer D	Intermediate Layer X
Primary Air Pressure	psi	1.6	6.3
Primary Air Temperature	°F	517	514
Forming Distance	in	7	18
Primary Air Flow	scfm		
Measured Variables:			
Die Tip Pressure	psi	79	117
Die Tip (Melt) Temperature (and Triton X-102 surfactant quench)	°F	495	514
Bonding:			
Pattern Roll Temperature			
Set Point	°F	220	
Actual Surface	°F		
Anvil Roll Temperature			
Set Point	°F	230	
Actual Surface	°F		
Pressure (Average)	psi	16	
Line Speed	fpm	22	

		3D Layer 55	3D Layer 207
Primary Air Pressure	psi		1.5
Primary Air Temperature	°F	545	522
Forming Distance	in		7
Primary Air Flow	scfm	400	195
Measured Variables:			
Die Tip Pressure	psi	174	56
Die Tip (Melt) Temperature (and Triton X-102 surfactant quench)	°F	538	502
Bonding:			
Pattern Roll Temperature			
Set Point	°F	180	237
Actual Surface	°F	160	207
Anvil Roll Temperature			
Set Point	°F	180	224
Actual Surface	°F	170	202
Pressure (Average)	psi	10	22
Line Speed	fpm	14	39

As with the previous examples, the data for these inventive samples was all normalized to a basis weight of 1 gsm. Both the actual data and the normalized data which show the characteristics of these inventive samples are set out in Table 4 below.

TABLE 4 -- COMPARISON OF COMPOSITES WITH 3D OUTER LAYERS

POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS IN THREE DIMENSIONAL MELTBLOWN

Sample Number	Composition	Basis Weight gsm	GRAB TENSILE Load MD g	Load CD g	Energy MD kg-mm	Energy CD kg-mm	Elong MD mm	Elong CD mm	TRAP TEAR Load MD g	Load CD g	NORMALIZED GRAB TENSILE Load MD g/gsm	Load CD g/gsm	NORMALIZED TRAP TEAR Load MD g/gsm	Load CD g/gsm
3D + x + 3D														
53	30 = 60:40 PE:PP x = 60:40 PULP:EVA	80	1895	1960	85	98	69	80	539	647	24	25	7	8
56	3D = 75:25 PE:PP x = 60:40 PULP:EVA	103	1651	1878	39	77	26	40	522	270	16	18	5	3
213	3D = 75:25 PP:PB x = 70:30 PULP:EVA	88	2301	2117	145	161	85	97	828	816	26	24	9	9
C	3D = 80:20 PP:PB 3D x = 30:70 PULP: (85:15 PP:PB)	111	5269	4272	175	182	44	63	1221	653	47	38	11	6
D	3D = 80:20 PP:PB 3D x = 30:70 PULP: (85:15 PP:PB)	112	4217	3314	117	108	38	46	980	648	38	30	9	6
3D + 3D (no intermediate layer)														
55	3D = 75:25 PE:PP	76	2118	1689	63	32	31	29	573	226	28	22	8	3
207	3D = 80:20 PP:PB	50	2334	1856	111	108	71	89	892	661	47	37	18	13

It can be seen from Table 4 that the laminate material formed from polyethylene, namely samples 53 and 56, do not exhibit the same strength, toughness, and tear resistance as those formed from polybutylene, namely Samples 213, C, and D. In particular, the polybutylene laminate materials bonded to an intermediate layer have an increase in peak load in the machine direction of 121% on average over the polyethylene laminate materials. Additionally, those polybutylene laminate materials have an increase in peak energy in the machine direction of 135% on average over the polyethylene laminate materials.

Accordingly, the inventive laminate materials containing polybutylene. Samples 213. C. and D demonstrate the increased strength and toughness of the three dimensional nonwoven webs prepared in accordance with the present invention.

As can also be seen from Table 4, Samples 213, C, and D formed with polybutylene, show much greater tear resistance than the samples formed with polyethylene.

It can also be seen from Table 4 that the three dimensional layer formed with polybutylene, Sample 207, had a 10% greater peak load than the three dimensional layer formed with polyethylene, Sample 55. As for peak energy, Sample 207 was 76% greater than Sample 55. Additionally, Sample 207's tear resistance was 56% greater than the tear resistance of Sample 55. It should be noted that there was no intermediate layer bonded between the two three dimensional layers of Samples 207 and 55.

Claims

1. A three dimensional nonwoven web comprising at least one layer consisting of drawn and unoriented thermoplastic fibers formed from a blend comprising polypropylene and polybutylene, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene.

2. A three dimensional nonwoven wiper comprising a web consisting of drawn thermoplastic fibers, wherein the fibers are formed from a blend comprising polypropylene and polybutylene to form thermoplastic fibers wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene.

3. A laminate material comprising at least one three dimensional layer consisting of drawn and unoriented thermoplastic fibers formed from a blend comprising polyproplene and polybutylene, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene.

4. A three dimensional nonwoven wiper comprising a laminate material comprising at least one three dimensional layer consisting of drawn and unoriented thermoplastic fibers formed from a blend comprising polypropylene and polybutylene, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene.

5. The material of one of the preceding claims, wherein the polypropylene is selected from the group consisting of poylpropylene homopolymers and polypropylene copolymers and the polybutylene is selected from the group consisting of polybutylene homopolymers and polybutylene copolymers.

6. The material of claim 5, wherein the polypropylene copolymer and the polybutylene copolymers are copolymers of ethylene.

7. A method for forming a layer of three dimensional nonwoven web consisting of thermoplastic fibers, comprising the steps of:
   a. mixing polypropylene and polybutylene to form a blend, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene;
   b. heating the blend to form a melt;
   c. extruding the melt through a die to form thermoplastic fibers;
   d. initially drawing the fibers to a ratio of greater than 14 to 1; and
   e. depositing the fibers onto a forming surface to form a first layer.

8. A method of forming a three dimensional nonwoven wiper consisting of thermoplastic fibers comprising the steps:
   a. mixing polypropylene and polybutylene to form a blend, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene;
   b. heating the blend to form a melt;
   c. extruding the melt through a die to form thermoplastic fibers;
   d. initially drawing the fibers to a ratio of greater than 14 to 1; and
   e. depositing the fibers onto a forming surface to form a first layer.

9. A method for forming a laminate material comprising at least one three dimensional layer consisting of thermoplastic fibers, comprising the steps of:

a. mixing polypropylene and polybutylene to form a blend, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene;
b. heating the blend to form a melt;
c. extruding the melt through a die to form thermoplastic fibers;
d. initially drawing the fibers to a ratio of greater than 14 to 1; and
e. depositing the fibers onto a forming surface to form a first layer.

10. The method of one of claims 7 to 9, wherein the fibers are quenched prior to being deposited on the forming surface.

11. The method of one of claims 7 to 10, wherein after the fibers are deposited, the layer or the wiper or the first layer is bonded.

12. The method of one of claims 7 to 11, wherein the polypropylene is selected from the group consisting of polypropylene homopolymers and polypropylene copolymers and the polybutylene is selected from the group consisting of polybutylene homopylmers and polybutylene copolymers.

13. The method of claim 12, wherein the polypropylene copolymer and the polybutylene copolymers are copolymers of ethylene.

14. The method of one of claims 9 to 13, wherein the first layer is bonded to a second layer selected from the group of materials consisting of foam, meltblown, coform, spunbound, scrim, and three dimensional meltblown.

15. The method of claim 14, wherein the second layer is bonded to a third layer, preferably selected from the group of materials consisting of foam, meltblown, coform, spunbound, scrim and three dimensional meltblown.

16. A three dimensional nonwoven web comprising at least one layer consisting of drawn thermoplastic fibers formed from a ternary blend comprising polypropylene, polybutylene, and a terpolymer, wherein the ternary blend by weight is from 10% to 30% polybutylene.

17. A three dimensional nonwoven wiper consisting of drawn thermoplastic fibers formed from a ternary blend comprising polypropylene, polybutylene, and a terpolymer wherein the ternary blend by weight is from 10% to 30% polybutylene.

18. A laminate material comprising at least one three dimensional layer consisting of drawn thermoplastic fibers formed from a ternary blend comprising polypropylene, polybutylene, and a terpolymer, wherein the ternary blend by weighs is from 10% to 30% polybutylene.

19. A three dimensional nonwoven wiper comprising a laminate material comprising at least one three dimensional layer consisting of drawn thermoplastic fibers formed from a ternary blend comprising polypropylene, polybutylene, and a terpolymer, wherein the ternary blend by weight is from 10% to 30% polybutylene.

20. The material of one of claims 17 to 19, wherein the terpolymer comprises propylene, ethylene, and 1-butene.

21. The material of one of claims 17 to 19, wherein the polypropylene is selected from the group consisting of polypropylene homopolymers and polypropylene copolymers and the polybutylene is selected from the group consisting of polybutylene homopolymers and polybutylene copolymers.

22. The material of claim 21, wherein the polypropylene copolymer and the polybutylene copolymer are copolymers of ethylene.

23. A method for forming a layer of three dimensional nonwoven web consisting of thermoplastic fibers, comprising the steps of:
a. mixing polypropylene, polybutylene, and a terpolymer to form a ternary blend, wherein the ternary

blend by weight is from 10% to 30% polybutylene;
b. heating the ternary blend to form a melt;
c. extruding the melt through a die to form thermoplastic fibers;
d. initially drawing the fibers to a ratio of greater than 14 to 1; and
e. depositing the fibers onto a forming surface to form a first layer.

24. A method for forming a three dimensional nonwoven wiper consisting of thermoplastic fibers comprising the steps of:
a. mixing polypropylene, polybutylene, and a terpolymer to form a ternary blend, wherein the ternary blend by weight is from 10% to 30% polybutylene;
b. heating the ternary blend to form a melt;
c. extruding the melt through a die to form thermoplastic fibers;
d. initially drawing the fibers to a ratio of greater than 14 to 1;
e. depositing the fibers onto a forming surface to form a first layer.

25. A method for forming a laminate material comprising at least one three dimensional layer consisting of thermoplastic fibers, comprising the steps of:
a. mixing polypropylene, polybutylene, and a terpolymer to form a ternary blend, wherein the ternary blend by weight is from 10% to 30% polybutylene;
b. heating the ternary blend to form a melt;
c. extruding the melt through a die to form thermoplastic fibers;
d. initially drawing the fibers to a ratio of greater than 14 to 1; and
e. depositing the fibers onto a forming surface to form a first layer.

26. The method of one of claims 23 to 25, wherein the terpolymer comprises propylene, ethylene and 1-butene.

27. The method of one of claims 23 to 26, wherein the polypropylene is selected from the group consisting of polypropylene homopolymers and polypropylene copoylmers and the polybutylene is selected from the group consisting of polybutylene homopolymers and polybutylene copolymers.

28. The method of claim 27, wherein the polypropiyene copolymer and the polybutylene copolymer are copolymers of ethylene.

29. The method of one of claims 23 to 28, wherein the fibers are quenched prior to being deposited on the forming surface.

30. The method of one of claims 23 to 29, wherein after the fibers are deposited, the layer or wiper or first layer is bonded.

31. The method of one of claims 25 to 30, wherein the first layer is bonded to a second layer selected from the group of materials consisting of foam, meltblown, coform spunbound, scrim and three dimensional meltblown.

32. The method of claim 31, wherein the second layer is bonded to a third layers preferably selected from the group of materials consisting of foam, meltblown, coform, spunbound, scrim and three dimensional meltblown.

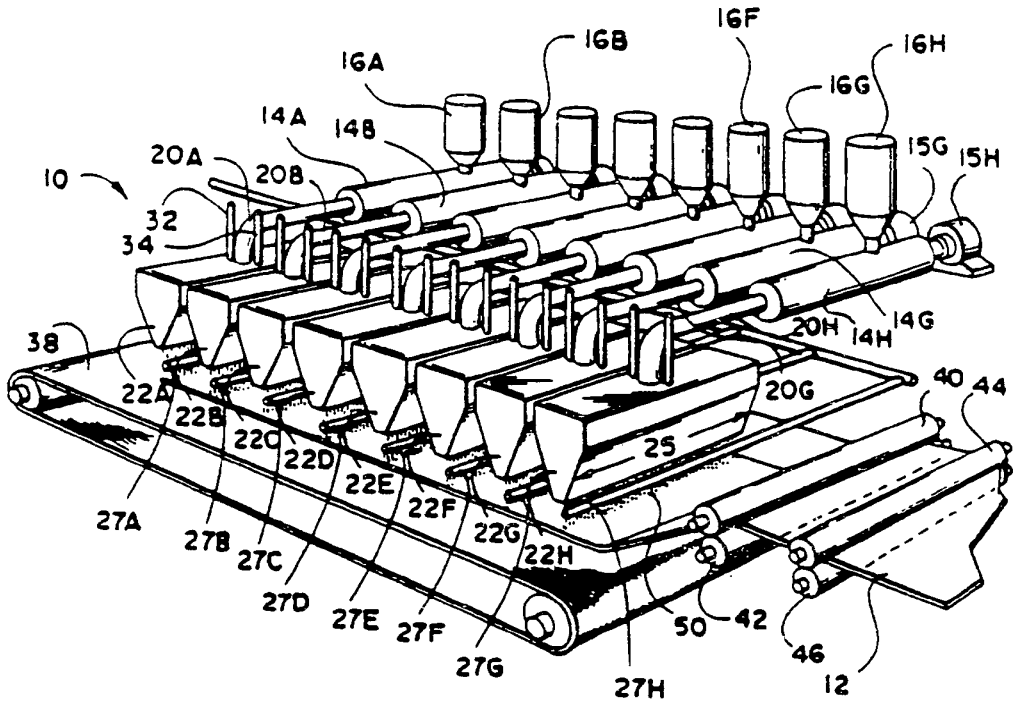

Fig_1

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 456 044 A3**

(12) **EUROPEAN PATENT APPLICATION**

(21) Application number: **91106709.8**

(51) Int. Cl.5: **D04H 1/42**

(22) Date of filing: **25.04.91**

(30) Priority: **04.05.90 US 518904**
13.12.90 US 626817

(43) Date of publication of application:
13.11.91 Bulletin 91/46

(84) Designated Contracting States:
AT BE CH DE DK ES FR GB GR IT LI LU NL SE

(88) Date of deferred publication of the search report:
04.12.91 Bulletin 91/49

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(54) **Three dimensional polymer webs and methods of making same.**

(57) Three-dimensional nonwoven webs comprise at least one layer consisting of drawn and unoriented thermoplatic fibers formed from a blend of polypropylene and polybutylene, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene. The blend can be a blend of a homopolymer of polypropylene and a homopolymer of polybutylene: a homopolymer of polypropylene and a copolymer of polybutlene: a copolymer of polypropylene and a homopolymer of polybutylene: and a copolymer of polypropylene and a copolymer of polybutylene. In addition, ternary blends comprising polypropylene, polybutylene and a terpolymer (propylene, ethylene, and l-butene) are disclosed. The resulting nonwoven webs have high strength, toughness, and tear resistance.

EP 0 456 044 A3

European Patent Office

EUROPEAN SEARCH REPORT

Application Number EP 91106709.8

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	EP - A2 - 0 277 710 (EXXON CHEMICAL PATENTS INC) * Claims 1,2,3,7 * --	1,2,3, 7,8,9	D 04 H 1/42
A	EP - A2 - 0 138 549 (MINNESOTA MINING AND MANUFACTURING COMPANY) * Example 1 * ----	7	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

D 04 H 1/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	04-10-1991	KAMMERER

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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EPO FORM 1503 03.82 (P0401)